

## **Appendix F**

### **Sample Documentation and Shipment Instructions**

#### **F.1 Documentation**

F.1.1 Scope and application. This section describes procedures for maintaining sample control through proper sample documentation. When samples are collected for chemical or physical characteristics analysis, documentation such as sample labels, daily contractor quality control reports (QCR), chain-of-custody and sample analysis request forms, custody seals, and field logbooks need to be completed. The information presented in this section enables maintenance of sample integrity from time of sample collection through transportation and storage. It is this documentation that will verify that samples were handled properly.

F.1.2 Documentation. The following discussion outlines standard practices and procedures to be used when documenting a sampling episode. All project-specific documentation requirements must be presented in the sampling and analysis plan (SAP). This includes identification of procedures required for field documentation, sample labeling, and the maintenance of chain-of-custody. Applicable requirements are identified in the following sections. Proper completion of all documentation with indelible ink is necessary to support the use of these records in any potential enforcement actions that may result. Protocols for corrections to documentation should not obliterate data entries, but place a single line through incorrect entry, noting corrected information, recorder's initials, and date correction was performed. Maintaining sample integrity through proper documentation is essential. Following site activities, all project documentation becomes a part of the final evidence file. These records should be maintained for a certain period of retention time. The documentation retention time requirements of a project must be presented within the SAP and may be based on the use of the data, funding source, or regulatory authority.

F.1.2.1 Daily contractor quality control reports (QCR). During the field investigation or remedial action activities, daily contractor QCRs should be prepared daily, dated, signed by the project contractor quality control representative, and sent to USACE at a rate specified in the scope of work or specifications. With respect to geotechnical and chemical procedures, these reports should include weather information at the time of sampling, field instrument measurements, calibrations, identification of all field and control samples taken, departures from the approved SAP necessary, deviations from approved geotechnical procedures (such as well installation or drilling), any problems encountered, and instructions from Government personnel. Any deviations that may affect data quality objectives must be conveyed to U.S. Army Corps of Engineers (USACE) personnel (technical manager, project geologist, project chemist, etc.) immediately. The following should be attached to the daily contractor QCRs: quality assurance (QA) sample tables that match up primary, replicate (quality control (QC)/QA), and other field control samples (e.g., blanks), copies of chain-of-custody forms, field-generated analytical results, and any other project forms that are generated. Additional documentation requirements of the daily contractor QCRs are outlined in Engineer Regulation (ER) 415-1-302 and Corps of Engineers Guide Specification (CEGS) 01451.

F.1.2.2 Field logbooks. Sampling situations vary widely. No general rules can specify the exact information that must be entered in a field logbook for a particular site. However, the logbook should contain sufficient information to enable the sampling activity to be reconstructed without relying on the collector's memory. Project field logbooks should be bound and have numbered, water-resistant pages. Record the site name and project name and number on inside front cover of logbook. All pertinent information regarding the site and sampling procedures must be documented as near to real-time as possible. At the conclusion of each day, the person maintaining the logbook should sign and date the day's

documentation entries. Notations should be made in logbook fashion, noting the time and date of all entries. Information recorded in other project documents (e.g., boring logs, well installation/development logs, or drum logs) should not be repeated in the field logbook, except in summary form to avoid transcription errors. Logbooks should be kept in the field team member's possession or in a secure place during field work. Following site activities or if the logbook is completely filled, the logbook becomes a part of the project final evidence file as noted previously. The technical planning team may also elect to establish documentation requirements that follow a more uniform organization than a field logbook. These documentation requirements would include the use of project forms. This approach helps enhance consistency in the information recorded and streamlines the documenting process. Any forms proposed for use should be task specific and should incorporate appropriate topics from those identified as follows. All forms must be presented in the project SAP. The following are some suggested topics to include in the field logbook:

- Name and exact location of site of investigation or interest.
- Name and title of person maintaining logbook (author).
- Date and time of arrival and departure at site location.
- Purpose of site visit or sampling activity.
- Name and address of field contact. This may also include information on access agreements.
- Names and responsibilities of all persons on site.
- Names, affiliations, and purpose of all site visitors.
- Level of personal protective equipment worn at the site.
- Weather conditions on the day of sampling, and any additional environmental conditions or observations pertinent to field activities.
- Field instrumentation or equipment used, and purpose of use (i.e., health and safety screening, sample selection for laboratory analysis). Note source, quality, or lot numbers for any supplies or reagents (e.g., sample containers, preservatives, reagents, water for field blanks/field control samples, and decontamination procedures). Retain any certificates or information supplied with the equipment used.
- Type of waste, suspected waste concentrations if known, and sample matrices to be handled.
- Document the sample collection method and any sample handling procedures such as filtration, compositing, and executed preservation techniques used.
- Document the sample location. If a compositing scheme is used, clearly identify appropriate locations for all sample aliquots included within each composite sample. Prepare a dimensional sketch of the general surroundings of the sampling area (site), and/or support with other forms of documentation (i.e., photographic log). Sample identification numbers should correspond directly with sample locations.

- Identify sample numbers, volumes, and containers (number, size, type) used for each sample collected. Note the date and time of each sample, identify any associated QC samples, or any factors that may affect the quality.
- Record any field measurements, field screening/analytical results generated, calibration methods used, field results, and QC information.
- Identify decontamination procedures employed for sampling equipment.
- Document appropriate references to maps and photographic logs of the sampling site.
- Record information on scheduling modifications, change orders, sampling or drilling decisions/changes.
- Describe the number of shipping coolers packed, note chain-of-custody (COC) numbers or attach a copy of COC, and record the mode of transportation and applicable tracking numbers.
- Record name and address of all receiving laboratories.
- Maintain appropriate documentation for investigation-derived wastes. Note contents and volumes of waste generated, storage, and disposal methods used.

F.1.2.3 Documenting sampling points and locations. The exact locations of sampling points should be documented for purposes of generating an accurate representation of the site conditions using the data generated to date, defining data gaps, and identifying potential future data needs. A monument should be chosen at each site to act as a stationary reference point from which all sampling points can be measured using a compass and measuring tape. If a building or other stationary structure exists, its corner may act as this reference point. If no monument exists, it will be necessary to create one. A piece of wood, approximately 5 cm by 5 cm (2 in. by 2 in.), should be hammered into the ground to almost ground level, making it difficult to remove and thus assuring its permanence. The stake should then be marked with flagging tape or fluorescent paint. When applicable, sampling points associated with coordinates that are referenced to a position on the earth must comply with ER 1110-1-8156. ER 1110-1-8156 requires geospatial data to be documented using the Federal Geographic Data Committee's content standards for digital geospatial metadata. Geospatial data are nontactical data, referenced either directly or indirectly to a location and boundaries on the earth. Additional guidance on geospatial data systems may be found in EM 1110-1-2909. To establish a sampling point, the following procedure is recommended:

- Standing at the monument, facing the sampling point, use the compass hairlines to determine the degree of direction.
- Ensure that the line of sight runs from the monument, through both hairline needles on the compass, to the sampling point.
- When first establishing the sampling point, record the degree and direction reading from the compass in the field logbook, along with the distance measurement from the monument to the sampling point.

F.1.2.4 Photographic documentation. All sampling points should be documented on film. A film record of a sampling event allows positive identification of the sampling point. In some cases, a photograph

of the actual sample collected may also be required. Photographs are the most accurate and convenient record of field personnel observations. Photographs taken to document sampling points should include two or more reference points to facilitate relocating the point at a later date. Keeping a record of photographs taken is crucial to their validity as a representation of an existing situation. Photographic documentation is invaluable if the sampling and subsequent analytical data end in litigation, enforcement, or cost recovery actions. In addition to photographs, video coverage of a sampling episode can be equally as valuable as or even more valuable than photographs because it can be used to prove that samples were taken properly as well as verify the location at which they were taken. Video coverage can be used as a record of site conditions and can give those who have not been onsite an idea of the circumstances. For each photograph taken, the following items should be noted in the field logbook:

- Date.
- Time.
- Photographer (signature).
- Name of site.
- General direction faced and description of the subject.
- Sequential number of the photograph and the roll number.
- Site photo map (see Figure F-1).

#### F.1.2.5 Sample documentation

F.1.2.5.1 Sample labels. Sample labels are required for properly identifying samples and evidence. All samples must be properly labeled with the label affixed to the container prior to transportation to the laboratory. It is also recommended that samples be photographed so that labels are clearly readable for later identification. Information on sample labels should include, but not be limited to, the following:

- C Project Code. An assigned contractor, project number, site name.
- C Station Number. A unique identifier assigned to a sampling point by the sampling team.
- C Sample Identification Number. Each sample, including field control samples, collected for a project should be assigned a unique number. This assigned number incorporates information on the sample type and date as noted in Section F.1.2.5.2.
- C Samplers. Each sampler's name and signature or initials.

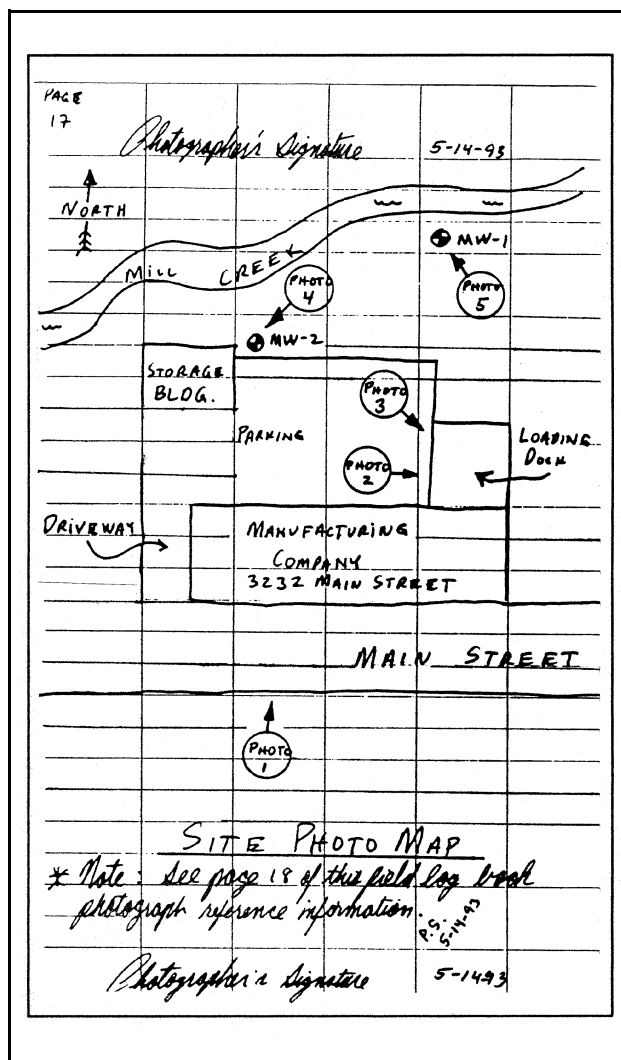


Figure F-1. Site photo map

- C Preservative. Whether a preservative is used and the type of preservative.
- C Analysis. The type of analysis requested.
- C Date/Time. Identify the date and time the sample was taken.
- C Type of Sample. The type of sample should be identified as discrete or composite.

F.1.2.5.2 Sample numbering. A sample numbering system should be used to identify each sample collected and submitted for analysis. The purpose of the numbering system is to assist in the tracking of samples and to facilitate retrieval of analytical results. The sample identification numbers for each sampling effort should be used on sample labels, sample tracking matrix forms, COC forms, field logbooks, and all other applicable documentation. A listing of all sample identification numbers should be recorded in the field logbook. The sampling numbering system may vary depending upon the number and type of samples that will be collected at the site. An example of a sample numbering system follows. Location and sample identification numbers should consist of the following designations to identify the location (AABBB-CC), sample sequence number, date (MMDDYY), and sample depth interval for soils (00-00):

- For soil: AABBB-CC/MMDDYY/00-00
- For water: AABBB-CC/MMDDYY
- For QC samples: AABBB-CC/MMDDYY

Example: SB001-01/081492/08-10 = Soil Boring SB001 Sample Number 1, sampled on August 14, 1992, from a sample depth interval of 8 to 10 ft (2.4 to 3 m). Duplicate samples should be numbered in sequential order. For example, a duplicate sample collected from this soil boring example would have a designation as follows: SB001-02/081492/08-10. Each sample collected must be assigned a unique sample number. Sample numbers should change when the media or location changes. Sample numbers should not change because different analyses are requested. For example, water samples collected at the same location, date, and time for volatile organics, semivolatile organics, and metals analyses would all have the same sample number, although the various sample aliquots would be collected in different containers.

F.1.2.5.3 Chain-of-custody (COC). COC procedures provide documentation of the handling of each sample from the time it is collected until it is destroyed. COC procedures are implemented so that a record of sample collection, transfer of samples between personnel, sample shipping, and receipt by the laboratory that will analyze the sample is maintained. Records concerning the cleaning of empty sample containers, container shipment from the laboratory to the site, and security of empty containers at the site should also be maintained. The COC record (Figure F-2) serves as a legal record of possession of the sample. The COC record is initiated with the acquisition of the sample. The COC record remains with the sample at all times and bears the name of the person (field investigator) assuming responsibility for the samples. The field investigator is tasked with ensuring secure and appropriate handling of the bottles and samples. To simplify the COC record and eliminate potential litigation problems, as few people as possible should handle the sample or physical evidence during the investigation. A sample is considered to be under custody if one or more of the following criteria are met:

[illegible]

**Figure F-2. Chain-of-custody form**

C

- ☐ The sample is in the sampler's possession.
- ☐ The sample is in the sampler's view after being in possession.
- ☐ The sample was in the sampler's possession and then was locked up to prevent tampering.
- ☐ The sample is in a designated secure area.

In addition to the COC record, there is also a COC (custody) seal. The COC seal (Figure F-3) is an adhesive seal placed in areas such that if a sealed container is opened, the seal would be broken. The COC seal ensures that no sample tampering occurred between the field and the laboratory analysis.

**F.1.2.5.4 Transfer of custody and shipment.** All sample sets should be accompanied by a COC record. When transferring possession of samples, the individual receiving the samples should sign, date, and note the time that he/she received the samples on the COC record. This COC record documents transfer of custody of samples from the field investigator to another person, other laboratories, or other organizational units. Samples must be properly packaged for shipment and delivered or shipped to the designated laboratory

for analyses. Shipping containers must be secured by using nylon strapping tape and custody seals (Instruction F-2). The custody seals must be placed on the container so that it cannot be opened without breaking the seals. The seal must be signed and dated by the field investigator. When samples are split with a facility, state regulatory agency, or other government agency, the agency representative must sign the COC record, if present. All samples should be accompanied by the COC record. The USACE tracking number (e.g., laboratory information management system (LIMS) number) that is used in conjunction with the

<p>CUSTODY SEAL</p>  <p>_____</p> <p>Date</p> <p>_____</p> <p>Signature</p>	<p>CUSTODY SEAL</p>  <p>_____</p> <p>Date</p> <p>_____</p> <p>Signature</p>
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**Figure F-3. Chain-of-custody seal**

Government QA sample shipment must be written on the COC record of the QA sample. The original and one copy of the record will be placed in a plastic bag taped to the inside lid of the secured shipping container. One copy of the record will be retained by the field investigator or project leader. The original record will be transmitted to the field investigator or project leader after samples are accepted by the laboratory. This copy will become a part of the project file. If sent by mail, the package should be registered with return receipt requested. If sent by common carrier, an air bill should be used. Receipts from post offices and air bills should be retained as part of the documentation of the COC. The air bill number or registered mail serial number should be recorded in the remarks section of the COC record.

**F.1.2.5.5 Sample analysis request.** To ensure that proper analysis is performed on the samples, additional paperwork may need to be filled out, as required by the laboratory performing the analysis. This form identifies samples by number, location, and time collected and allows the collector to indicate the desired analysis. This form should act as a supplement/confirmation to the COC record and laboratory contacts made prior to the sample event initiation.

### F.1.3 QA/QC requirements.

**F.1.3.1 Corrections to documentation.** All original data recorded in field logbooks and on sample labels, COC records, and receipt-for-samples forms are written in waterproof ink. If an error is made on an accountable document, corrections should be made simply by crossing out the error and entering the correct information. The erroneous information should not be obliterated. Any error discovered on a document should be corrected by the person who made the entry. All corrections must be initialed and dated.

**F.1.3.2 Photographs.** The photographer should review the photographs or slides when they return from developing and compare them with the photographic log to confirm that the log and photographs match.

**F.1.4 Potential problems.** Although most sample labels are made with water-resistant paper and are filled out using waterproof ink, inclement weather and general field conditions can affect the legibility of sample labels. It is recommended that after sample labels are filled out and affixed to the sample container, the label should be covered with wide clear tape. This will preserve the label and keep it from becoming illegible. In addition to label protection, COC and analysis request forms should be protected when samples are shipped in iced coolers. Typically, these forms should be placed inside a ziplock bag or similar waterproof protection and taped to the inside lid of the secured shipping container with the samples.

## F. 2 Packaging and Shipping Procedures

F.2.1 Scope and application. This section describes procedures for properly packaging and shipping environmental and hazardous waste samples, as well as the shipment of preservatives used in the environmental sampling. Guidelines for proper container and preservative selection can be found in Appendix B. Personnel that are involved in packaging, shipping, and receipt of samples must be aware of Department of Transportation (DOT) regulations, know when to apply them, and know what procedures are needed to support this application. Personnel who ship samples considered a DOT hazardous material (HM) must be trained in accordance with the requirements set in 49 CFR 172.704. The following procedures identify packaging and shipping requirements for environmental and hazardous waste samples that are both DOT-regulated and nonregulated. Further information is presented for shipping carriers (i.e., FedEx, UPS) that use the International Air Transportation Association (IATA) regulations to govern domestic and international shipments. Shipping procedures for common preservatives and decontamination fluids are also addressed. Finally, it should be noted that DOT regulations also apply to the shipment of asbestos samples.

F.2.2 Procedures for shipping environmental samples. Environmental samples are defined as those samples collected from environmental matrices such as soil, groundwater, or sediments. The following sections identify packaging and shipping requirements for environmental samples that are unpreserved or preserved by acid/base/chemical addition. The following general procedures apply to the packaging of all environmental samples:

- Verify that the sample label is complete and adequately identifies the items described in Instruction F-1.
- Verify that each sample cap/lid is secured on the bottle, and place each sample in a plastic bag. For multiple volatile organic analysis (VOA) vials, all vials from each sample location should be placed in a small plastic bag at a minimum. Evidence tape or custody seals may be placed over the sample lid and container, or over the seal of the bag for additional security, if desired.
- Squeeze as much air as possible from the bag, and seal the bag. Trip blanks are packaged in the same manner as that for aqueous VOA samples.
- Prepare the shipping container for use. For a commercial cooler, this includes taping the drain plug shut inside and out, and lining the cooler with a large plastic garbage bag. Place approximately 7.5 cm (3 in.) of inert packing material in the bottom of the liner. Place vermiculite or perlite on the bottom if the materials are liquid. Alternative shipping containers may be used if approved by project technical personnel.
- Place the samples upright in the lined cooler or storage container in such a way that the samples will not touch each other during shipment. Add inert packing material as necessary to ensure separation of samples.
- With the exception of aqueous metals analyses, all environmental samples should be shipped to the laboratory on ice and chilled to  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . If any of the shipped samples require cooling, place double bags of ice around the containers. Also include a 40-mL VOA vial filled with water for use as a temperature blank for the laboratory.



- Fill the cooler with packing material and tape the inner liner shut. NOTE: Do not use “environmentally friendly” peanuts made of starch to pack containers of liquids. These packing materials will dissolve when they get wet or moist.
- Place the paperwork (Instruction F-1) being sent to the laboratory inside a plastic bag and tape it to the inside of the cooler lid. Include a copy of the COC form in the paperwork sent to the laboratory. The sampler keeps one copy of the COC form. Include any additional paperwork to notify the laboratory of project information (laboratory notification checklist), or if a sample is suspected of containing any substance for which laboratory personnel should take safety precautions.
- Close the cooler and seal it with strapping tape.
- Place at least two custody seals on the outside of the cooler (one on the front and one on the back). More custody seals may be used at the discretion of the sampler.
- Prepare standard air bill paperwork for shipment of the samples to the laboratory. Personnel should be aware of carrier weight or other policy restrictions.

F.2.2.1 Unpreserved environmental samples. Environmental samples that are shipped unpreserved are not considered a hazardous material by DOT if they do not exhibit a DOT hazard class. This exempts them from DOT regulation during transportation to the laboratory. In general, this applies to soil/sediment and aqueous samples preserved by cooling. Follow the general procedures identified in Section F.2.2 for packaging and shipment of unpreserved environmental samples.

F.2.2.2 Preserved environmental samples. All samples that are preserved by the addition of chemicals are subjected to greater scrutiny when defining whether DOT regulations apply and the appropriate packaging and shipping requirements necessary. Care should be exercised when adding any chemicals to environmental samples for preservation purposes. Samples must be observed, noting any chemical reactions that take place, and following with any contingency measures needed to obtain a representative sample. Most importantly, add only the amount of preservative needed to achieve the required preservation criteria. Excessive preservation may make an otherwise unregulated sample subject to DOT hazardous materials regulations. On average, testing of water samples arriving at commercial laboratories has shown that between 30 and 50 percent of the samples are excessively preserved and may have been improperly packaged and shipped. Samples in this category may also be considered a Resource Conservation and Recovery Act (RCRA) characteristic or listed waste. However, an exclusion from manifesting and hazardous waste marking requirements exists under RCRA for shipping environmental samples to the laboratory. The RCRA exclusion can be found in 40 CFR 261.4(d). Materials that are Toxic Substances Control Act (TSCA) regulated (polychlorinated biphenyls (PCBs)) may be shipped under exclusions found in 40 CFR 761.65(I). However, these exemptions do not pertain to DOT regulations, and compliance with DOT regulation is still required. Field personnel should also realize that even though the DOT may allow for these items to be shipped by air, other international regulations governing air carriers (such as IATA/International Civil Aviation Organization (ICAO)) may further limit or forbid materials allowed by DOT. The shipper must also comply with all packaging regulations. These regulations will be found in 49 CFR 171.101 and 49 CFR 173. Packaging and shipping requirements for preserved environmental samples are determined based on whether the preserved samples exhibit the same physical or chemical characteristics that initially defined the preservative as a DOT hazardous material. Follow the guidance presented in Section F.2.2.2.1 and in Sections F.2.2 or F.2.3 to determine the appropriate shipping and packaging requirements to apply.

F.2.2.2.1 Excepted preserved environmental samples. The DOT definition of a corrosive material is based on the destruction of intact animal skin. The DOT packing group (PG) associated with these samples determines the packaging and shipping procedures necessary, which are based on the time frame of observed tissue damage. Alternative testing to the animal testing has been developed and is accepted by DOT to support an application for exception from DOT classification and regulation. In the spring of 1997, USACE performed alternative testing to support the exception from the determination of acid/base preserved samples as a DOT corrosive classification. The testing showed that samples preserved in the manner described in Table F-1 and packaged as outlined in paragraph F.2.2 did not meet the definition of corrosive materials as prescribed by DOT under 49 CFR 173.136 (a)(1), 173.137 (a,b,c(1)). Based on this information, environmental samples meeting these criteria may be shipped as non-DOT regulated material following procedures outlined in Section F.2.2 under the USACE number DOT-E10904. Further information on this exception should be obtained from the USACE Hazardous, Toxic, Radioactive Waste - Center of Expertise (HTRW-CX) prior to its application or use. For consistency, the proper preparation of sample preservatives is outlined as follows for some common preservatives used in conjunction with environmental sampling:

**C Acid preservatives:**

- (1) Hydrochloric acid      500 mL water + 2.0 mL 12N hydrochloric acid
- (2) Nitric acid              500 mL water + 2.0 mL 15.7N nitric acid
- (3) Sulfuric acid            1000 mL water + 1.0 mL 18M sulfuric acid

• **Base preservatives:**

Sodium hydroxide              500 mL water + 1.0 mL 30% sodium hydroxide

Table F-1 identifies the required pH ranges for sample preservation to meet standard U.S. Environmental Protection Agency methods, and to avoid meeting the definition for a particular DOT hazard classification and packing group. Samples preserved in the field should be verified for correct pH to assess the utilization of this exception. This may become problematic if the testing materials used (i.e., pH test strips) do not distinguish pH values to the required accuracy. Based on the range limits established, it is mandatory to use pH paper capable of a minimum of 0.5 pH unit resolution, such as the use of short-range pH paper. However, due to the proximity of several of these values, it is recommended a pH meter be used to allow resolution to 0.1 pH unit. Field personnel are cautioned that, based on specific acid type, if pH is adjusted below the acid range limit or above the basic range limit, preserved samples will be regulated by DOT as a hazardous material and will require proper shipping papers, marking, and labeling as identified in Section F.2.2.2.2.

**Table F-1**  
**Preserved Sample pH Ranges Needed for DOT Exemption**

<b>Preservative Used</b>	<b>Acceptable Sample pH Ranges for DOT Exemption Status<sup>1</sup></b>
Hydrochloric acid	pH = 1.43 to 2.00
Nitric acid	pH = 1.33 to 2.00
Sulfuric acid	pH = 1.31 to 2.00
Sodium hydroxide	pH = 12.00 to 12.58

<sup>1</sup> Per USACE exemption DOT-E10904

F.2.2.2.2 Acid/base preserved environmental samples. Environmental samples that have been overpreserved may meet the DOT definition of hazardous materials (i.e., corrosive liquid). When applicable, these materials must be shipped in proper DOT-approved containers, unless the shipper uses limited-quantity exceptions, or if the shipper has determined through testing that the material does not meet the definition of a DOT corrosive (Section F.2.2.2.1). DOT and IATA regulations should be reviewed to determine appropriate packaging and shipping requirements, and if a limited-quantity exemption exists. If a limited-quantity exemption does exist, the volume of hazardous material to be shipped is evaluated to determine if it exceeds this threshold limit. Refer to Table F-2 for information on the proper shipping name (PSN) and the limited quantities associated with some common types of acid preservation. When the total volumes of shipped material are less than the limited quantities noted, then the materials may be shipped as a limited quantity following the sample packaging and shipment procedures included in Section F.2.3, with “Limited Quantity” or “Ltd Qty.” recorded on the shipping papers after the PSN. Table F-3 summarizes general requirements necessary to ship DOT-limited quantities. If the shipper does not meet these requirements, then the samples are considered fully regulated DOT hazardous material, and all DOT requirements as defined in 49 CFR 171-178 must be met.

**Table F-2**  
**PSN and Limited Quantities for Common Preservatives, Decontamination Fluids, and Potentially Hazardous Samples<sup>1</sup>**

Chemical Compound/Sample/ Material	PSN	Lmt Qty <sup>2</sup>	Lmt Qty <sup>3</sup>
Sulfuric acid >51%	Sulfuric Acid, with more than 51% sulfuric acid, 8, UN1830, PGII	1 liter	30 Liters
Sulfuric acid <51%	Sulfuric Acid, with not more than 51% sulfuric acid, 8, UN2796, PGII	1 liter	30 Liters
Nitric acid >70%	Nitric Acid, with more than 70% nitric acid, 8, UN2031, PGI	Forbidden	2.5 Liters
Nitric acid <70%	Nitric Acid, with less than 70% nitric acid, 8, UN2031, PGII	Forbidden	2.5 Liters
Nitric acid solution (10%) <sup>4</sup>	Nitric Acid, other than red fuming with less than 20% nitric acid, 8, UN2301, PGII	1 liter	30 liters
Nitric acid solution (1%) <sup>4</sup>	Nitric Acid, other than red fuming with less than 20% nitric acid, 8, UN2301, PGII	1 liter	30 liters
Nitric acid preserved samples	Corrosive Liquids, Acidic, Inorganic, NOS (Nitric Acid), 8, UN3264, PGIII	5 liters	60 liters
Hydrochloric acid	Hydrochloric Acid Solution, 8, UN1789, PGII	1 liter	30 liters
Hydrochloric acid preserved samples	Corrosive Liquids, Acidic, Inorganic, NOS (Hydrochloric Acid), 8, UN3264, PG III	5 liters	60 liters
Phosphoric acid	Phosphoric Acid, 8, UN1805, PGIII	5 liters	60 liters
Sodium hydroxide solution	Sodium Hydroxide Solution, 8, UN1824, PGII	1 liter	30 liters
Sodium hydroxide preserved sample	Corrosive Liquids, Basic, Inorganic, NOS (Sodium Hydroxide), 8, UN3266, PGIII	5 liters	60 liters
Isopropyl alcohol	Isopropyl alcohol or Isopropanol, 3, UN1219, PGII	5 liters	60 liters
Methanol	Methanol, 3, UN1230, PGII	1 liter	60 liters
Hexanes	Hexanes, 3, UN1208, PGII	5 liters	60 liters
Sodium bisulfate, aqu sol	Bisulfate, aqueous solution, 8, UN2837, PGII	1 Liter	30 liters
Fuel ID/Tank Samples	Flammable Liquids, NOS (fuels), 3, UN1993, PGIII.	60 liters	220 liters

<sup>1</sup> Personnel should be aware that the following represents only a few of the possible Proper Shipping Names (PSNs) available for shipping hazardous materials. Not all materials field personnel may have to ship are covered by these examples. Shippers are advised to review the DOT hazardous materials table prior to shipping.

<sup>2</sup> Passenger Aircraft/Railcar.

<sup>3</sup> Cargo Aircraft Only.

<sup>4</sup> Nitric acid solutions at #20% acid are recognized by the ICAO and IATA, and are addressed in 49 CFR 171.11.

**Table F-3**  
**Limited Quantity Packaging and Shipping Paper Criteria**

Parameter	Class 3	Class 6.1	Class 8	Class 9
Outer Package (max gross wt)	30 kg/66 lb	30 kg/66 lb	30 kg/66 lb	30 kg/66 lb
Outer Package Markings:				
1. PSN	/	/	/	/
2. UN#	/	/	/	/
3. Orientation Arrows - liquids	/	/	/	/
4. Address	/	/	/	/
5. RQ	(/)	(/)	(/)	(/)
6. Other Markings: Marine Pollutant, Cargo Air Craft, etc.	(/)	(/)	(/)	(/)
Label	<u>Air:</u> Flammable Liquid <u>Other:</u> excepted	<u>Air:</u> Poisonous <u>Other:</u> excepted	<u>Air:</u> Corrosive <u>Other:</u> excepted	<u>Air:</u> Class 9 <u>Other:</u> excepted
Inner Package Size Limit:				(Not PG specific)
1. PGI	0.5 L	N/A	N/A	4.0 L/5.0 kg
2. PGII	1.0 L	N/A	1.0 L/1.0 kg	
3. PGIII	5.0 L	4.0 L/5.0 kg	4.0 L/5.0 kg	
Shipping Paper Entries:				
1. Gross wt	/	/	/	/
2. Number of packages	/	/	/	/
3. Proper shipping description	/	/	/	/
4. "Ltd Qty"	/	/	/	/
5. Marine Pollutant	(/)	(/)	(/)	(/)
6. RQ	(/)	(/)	(/)	(/)
Note:				
/ Entry Required				
(/) Required if definition criteria are met				

F.2.2.2.3 Methanol preserved samples. Soil and sediment samples may require preservation with methanol if samples will be tested for volatile organics by the high-level method. In this case methanol is added neat to the sample and is, therefore, considered a hazardous material. DOT and IATA regulations should be reviewed to determine appropriate packaging and shipping requirements, and to determine if a limited-quantity exemption exists. If so, the volume of hazardous material to be shipped is evaluated to determine if it exceeds this threshold limit. Refer to Table F-2 for information on the PSN, and limited quantities associated with methanol. Additional information on limited quantities should be referenced from IATA regulations when applicable. Again, refer to Table F-3 for a summary of general requirements necessary to ship DOT-limited quantities. For instance if the total volume of methanol included within sample jars packaged in the shipping container does not exceed 1 L, then one may ship under the exception found in 49 CFR 173.150 (b)(2) for limited quantities. This is supported by the DOT Hazardous Materials Table, which identifies the methanol quantity limitation for passenger aircraft or railcar as 1 L (0.3 gal). When the total volumes of shipped material are less than the limited quantities noted, then the materials may be shipped as a limited quantity. "Limited Quantity" or "Ltd Qty." must be recorded on the shipping papers after the PSN if the exception is used. Sample packaging and shipment should follow procedures included in Section F.2.3. When greater than 1 L is included, sample packaging and shipment procedures noted within 49 CFR 172.701, Hazardous Materials Table, must be followed. Recommend packaging shipping containers to maintain the volume of methanol below the thresholds noted in Table F-2. Project personnel are also

encouraged to check with the shipping carrier used, to verify that additional, more stringent policy requirements do not exist for the shipment of flammable liquids such as methanol. Another option available under DOT is the small-quantity exception found in 49 CFR 173.4. Specifically, 49 CFR 173.4(a)(1)(i) states the maximum quantity of material per inner container is limited to 30 mL for authorized liquids, other than Division 6.1, Packing Group I materials (i.e., poisons). In other words, for Method 5035 (EPA/SW-846) preserved samples, if one has less than or equal to 30 mL of methanol or bisulfate aqueous solutions (sodium bisulfate) per inner (sample) container, this material is not subject to any other requirements of the hazardous materials regulations except those in 49 CFR 173.4. DOT hazard classes covered by this exception include Class 3, Division 4.1, Division 4.2 (PGII and PGIII), Division 4.3 (PGII and PGIII), Division 5.1, Division 5.2, Division 6.1, Class 7, Class 8, and Class 9. In addition to the 30-mL container limit, additional restrictions and requirements apply. Personnel taking this exception should review 49 CFR 173.4 carefully. Finally, no 49 CFR 173.21 (forbidden) materials may be packaged, the gross weight of the completed package cannot exceed 29 kg (64 lb), and the package cannot be opened or altered until it is no longer in commerce (transport). The shipper must certify conformance with the referenced sections by marking the outside of the package with the statement “*This package conforms to 49 CFR 173.4*” or alternatively until 1 October 2001 with the statement “*This package conforms to the conditions and limitations specified in 49 CFR 173.4.*” Further, the shipper must indicate on the air waybill under nature and quantity of goods, “*Dangerous goods in Excepted Quantities.*” The IATA also requires the application of an “Excepted Quantities” label. This label contains the certification language previously identified. Label entries include shipper signature, title, date, address, and indication of the hazard class and associated United Nations (UN) number.

F.2.2.2.4 Quantity limitations. One final restriction to note is that while 49 CFR 173.4 does not have a total net quantity limitation, IATA Dangerous Goods Regulations (DGR Section 2.7.5.2) does. For packing group II materials (i.e., methanol or sodium bisulfate) the total net quantity limit is 500 mL. This equates to 33 inner (sample) containers (i.e., VOA vials) containing up to 15 mL of preservative per outer package (cooler). When shipping DOT hazardous materials by air, shippers have additional restrictions that are identified in Columns 9A/9B of the 49 CFR 172.101 Hazardous Materials Table. Net quantity limits of methanol for passenger and cargo aircraft are 1 L and 60 L, respectively. The net quantity limits for sodium bisulfate solutions are 1 L and 30 L, respectively. Shippers should note that *these* quantities exceed the IATA small-quantity exception. Therefore, if the volume of preservative (methanol or sodium bisulfate solution) is kept less than 30 mL per inner (sample) container *and* total net quantity per outer package (cooler) is limited to 500 mL, then quantity limits given in DOT Hazardous Materials Regulations or IATA Dangerous Goods Regulations are not an issue provided packaging conforms with 49 CFR 173.4.

F.2.2.2.5 HTRW-CX assistance. The HTRW-CX has coordinated with the Logistics Support Activity Packaging, Storage, and Containerization Center at Tobyhanna Army Depot, Tobyhanna, PA, to develop a standard 49 CFR 173.4 tested and certified packaging. Materials needed to assemble these sample and shipping packages are readily available to field personnel from local hardware or retail stores. The protocol established is available for USACE personnel use by contacting HTRW-CX for additional information.

### F.2.3 Procedures for shipping hazardous samples.

F.2.3.1 Hazardous samples are defined as those that are typically highly contaminated, such as oils, sludges, discarded products, and items that exhibit a hazard as defined by DOT, or if it is suspected that they may be explosive, reactive, poisonous, toxic, flammable, or corrosive. Samples with visual evidence of explosives content (e.g., TNT flakes) should be considered suspect and managed appropriately. Hazardous waste samples taken for chemical analyses are normally taken in small volumes with preservation limited to cooling. Packaging and shipping requirements for hazardous samples are typically determined based on

any known contaminants or characteristics of the samples. In several cases, field screening techniques may be used to identify the packaging requirements necessary. The shipment of these samples to the laboratory may also be considered exempt from regulation under RCRA/TSCA as previously described and as referenced from 40 CFR 261.4(d) and 40 CFR 761.65(I). However, these exemptions do not pertain to DOT or IATA regulations. DOT-defined hazardous material samples must be packaged and shipped in accordance with all applicable DOT regulations, including those establishing sample container types and specifications, marking, labeling, placarding requirements, and the preparation of associated shipping papers. In most cases, the shipper will be able to package and ship these samples under the limited-quantity requirements found in column 8A, Packaging Exceptions, of the Hazardous Materials Table (49 CFR 172.101). If no limited-quantity exceptions are found in column 8A, then field personnel should use the following guidelines to determine appropriate packaging and shipping requirements.

F.2.3.2 Initially, the shipper must determine the appropriate DOT hazard class. If the shipper is unable to determine the proper DOT hazard class, due to the unknown nature of the sample, the shipper must consult 49 CFR 171-177 to determine the proper hazard class and shipping name. The next step is to look at column 8A or 8B to determine the proper outer and (if required) inner nonbulk packaging requirements and any applicable exceptions. As it is safest to assume that the materials to be shipped do meet the definitions of a DOT hazard class, the outer container must be properly labeled and marked and the shipper must comply with all regulations concerning shipping papers and placarding. The cooler, or other outer package if considered an overpack (49 CFR 171.8 and 49 CFR 173.25), must be marked and labeled accordingly. For air shipment of samples that meet the definition of a DOT-hazardous material, the shipper must also use the quantity limitations found in column 9 of the Hazardous Materials Table (49 CFR 172.101). Further recommend that the transportation by air be designated as cargo aircraft. By specifying cargo aircraft, the shipper is permitted to ship larger volumes of material in a single outer container with less stringent regulatory requirements. The requirements for packaging, packing, and shipping for hazardous samples are outlined as follows. (Note: The following protocol should be used for the shipment of hazardous samples only if the shipper is taking a limited-quantity exception, unless the “paint can” is a UN specification container (i.e., 1A2, 1B2, etc). If the shipper is not taking a limited-quantity exception, UN performance-oriented packing requirements apply. Do not assume paint cans are UN specification packages unless they are marked in accordance with 49 CFR 178.503.)

- Ensure sample container label is complete, and adequately identifies the items prescribed in Instruction F-1.
- Verify each sample cap/lid is secured onto the bottle. Tape shut the lid onto sample containers, and place each sample in a plastic bag. Activated carbon may also be placed with the sample within the plastic bag to prevent cross-contamination.
- Place evidence tape or custody seals over the sample lid and container, or over the seal of the bag for additional security, if desired.
- Squeeze as much air as possible from the bag, and seal the bag.
- Place each bottle upright in a separate paint can. Fill the paint can with vermiculite, and affix the lid to the can. The lid must be sealed with metal clips or with filament or evidence tape; if clips are used, the manufacturer typically recommends six clips.
- Place DOT Orientation arrows on the can to indicate which end is up.

- Mark each with the proper DOT shipping name and identification number for the sample. These can be found referenced in the Hazardous Materials Table. The information may be placed on stickers or printed legibly. A liquid sample of an uncertain/unknown nature is shipped as a flammable liquid with the shipping name "FLAMMABLE LIQUID, N.O.S." and the identification number "UN1993." A solid sample of uncertain nature is shipped as a flammable solid with the shipping name "FLAMMABLE SOLID, N.O.S." and the identification number "UN1325." If the nature of the sample is known, 49 CFR 171-177 is consulted to determine the proper marking, labeling and packaging requirements. Always use DOT-approved outer containers to ship samples that meet or are suspected to meet the definitions of a hazardous material.
- Place the cans upright in a cooler that has had its drain plug taped shut inside and out, and has been lined with a garbage bag. Place vermiculite or perlite on the bottom if the materials are liquid.
- All hazardous samples should be shipped to the laboratory on ice and chilled to  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .
- Place additional inert packing material (styrofoam peanuts) in the cooler to partially cover the sample bottles. If samples are required to be shipped to the laboratory with ice, place bags of ice around the containers. The cooler must then be filled with packing material and the inner liner taped shut. NOTE: Do NOT use "environmentally friendly" peanuts made of starch to pack containers of liquids. These packing materials will dissolve when they get wet or moist.
- Place the paperwork going to the laboratory inside a plastic bag and tape it to the inside of the cooler lid. A copy of the COC form should be included in the paperwork sent to the laboratory. The sampler keeps one copy of the COC form. The laboratory should be notified if a sample is suspected of containing any substance for which laboratory personnel should take safety precautions.
- Close the cooler and seal with strapping tape. Place at least two custody seals on the outside of the cooler (one on the front and one on the back). More custody seals may be used at the discretion of the sampler.
- Place the following markings on the top of the cooler:
  - (1) Proper shipping name (49 CFR 172.301).
  - (2) DOT UN/North America identification number (49 CFR 172.301).
  - (3) Shipper/consignee's name and address (49 CFR 172.301).
- Place the following labels on top of the cooler (49 CFR 172.406(e)):
  - (1) Appropriate hazard class label (adjacent to PSN).
  - (2) "Cargo Aircraft Only" (as needed, per 49 CFR 172.101).
  - (3) Certification statement: "Inside (inner) packages comply with the prescribed specifications"
- Place orientation markings on two opposite vertical sides indicating "This Way Up" in addition to the markings and labels described in preceding item (49 CFR 172.312).

- Use restricted-article air bills for shipment. The “Shipper Certification for Restricted Articles” section is filled out as follows:
  - (1) Number of packages or number of coolers
  - (2) Proper shipping name
  - (3) Classification
  - (4) Identification number
  - (5) Net quantity per package or per cooler
  - (6) Radioactive materials section (leave blank)
  - (7) Note passenger or cargo aircraft
  - (8) Name and title of shipper (printed)
  - (9) Emergency telephone contact number within 24 to 48 hr
  - (10) Shipper’s signature

IATA Dangerous Goods Regulations list the hazard classes for many compounds. If the materials to be shipped cannot be found on the list, it may be necessary to use a more generic (not otherwise specified (NOS)) description. IATA regulations apply mainly to international shipment of hazardous materials by air. However a number of overnight domestic carriers (such as FedEx and UPS) also use IATA regulations to govern domestic shipments. Quantity limitations concerning hazardous materials shipments are usually the same as DOT; however, exceptions exist. IATA regulations must be reviewed if a domestic carrier requires IATA quantity limitations. Examples of PSNs that may be appropriate for samples are included in Table F-2.

F.2.4 Procedures for shipping preservatives and decontamination fluids. Preservatives and decontamination solvents used in environmental and hazardous waste sampling are often hazardous materials. These materials must also be stored and shipped in accordance with all applicable regulations. The preferred method for transporting preservatives to the site, many of which are DOT hazardous materials, is to order them from a chemical supply company and have that company ship the materials directly to the sampling site or base of operations. This reduces the liability and regulatory compliance issues that must be dealt with for field personnel. If project personnel must ship the materials defined as DOT-hazardous for use as preservatives or decontamination fluids, compliance with all applicable DOT regulations, including proper shipping containers, container markings, placarding, packaging, labeling, and shipping paper requirements is required. When Government personnel will transport preservatives and decontamination fluids to the field in a Government vehicle via highway only, the less stringent 49 CFR 173.6, Materials of Trade Exceptions, may apply. Further information on the exception can be obtained from the HTRW-CX. Procedures for shipping preservatives and decontamination fluids are outlined as follows:

- Determine the proper DOT shipping name (PSN) for the materials to be shipped. The PSN for hazardous materials will be found in 49 CFR 172.101, Hazardous Materials Table. The PSNs for the most common preservatives are given in Table F-2.



- Determine whether there is an exception for limited quantities for the material(s) to be shipped. Refer to column 8A in 49 CFR 172.101, Hazardous Materials Table, under the PSN for the specific material that needs to be shipped.
- If there is a limited-quantity exception and the materials to be shipped meet those quantity limitations for the transportation methods shown in column 9A/9B of the Hazardous Materials Table and comply with the proper packaging requirements as shown in column 8A, then the materials may be shipped as a limited quantity. “Limited Quantity” or “Ltd Qty.” must be recorded on the shipping paper after the PSN if the exception is used. Some of the quantity limitations of the more common chemicals used as preservatives in environmental samples are given in Table F-2.
- If the material does not have an exception as a limited quantity, the next step is to determine the requirements to properly package the material under DOT regulations. Refer to 49 CFR 172.101, Column 8B. In this section (packaging authorizations for nonbulk packaging), a three-digit (\*\*\*) number is present. To find the proper section for packaging authorizations, see 49 CFR 173.(\*\*\*). For example, under Sulfuric Acid, the three-digit number found in column 8B is 202. So the shipper should look under 49 CFR 173.202 for the packaging authorizations for shipping sulfuric acid. The materials to be shipped **MUST** comply with the required packaging.
- Determine whether there are placarding requirements for the shipment. Refer to 49 CFR 172.500 for this information.
- Ship the materials following the criteria established.

#### F.2.5 Potential problems.

F.2.5.1 Field personnel should be aware that there are discrepancies for nitric acid in the shipping name tables for DOT and ICAO/IATA. IATA/ICAO allow the shipment of  $\leq 20$  percent nitric acid via passenger aircraft and allow these concentrations to be shipped as a limited quantity. DOT does not acknowledge this PSN entry, but does acknowledge ICAO technical instructions. Refer to 49 CFR 171.11 for additional details.

F.2.5.2 Note that excessive sample preservation is very likely to bring an environmental sample into the DOT hazardous materials regulatory realm. Depending on the specific inorganic acid used as a preservative, a difference of 0.5 pH unit (e.g., pH 1.0 versus pH 1.5) will likely trigger all DOT hazardous materials communication standards and regulations. The ranges noted in Table F-2 are provided to help field personnel make the appropriate decisions associated with the classification of preserved environmental samples for transportation.

F.2.5.3 Individual samples known to contain or are highly suspected of containing PCBs at greater than or equal to 0.45 kg (1 lb) or greater than or equal to 1 percent by weight are regulated in the air mode. In this instance DOT hazardous materials regulations are applicable. Further, PCBs are regulated as a marine pollutant, which requires additional notations on the shipping paper. Readers are referred to 49 CFR 172.203.